

Note

Efficient regeneration of aldehydes and ketones from their semicarbazones using sodium perborate: Part 4[†]

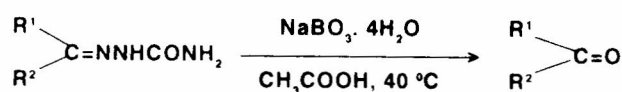
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Semicarbazones of aldehydes and ketones have been oxidized to the corresponding parent aldehydes and ketones in almost quantitative yields under mild conditions by using inexpensive sodium perborate.

Semicarbazones are used not only for purification of carbonyl compounds but also to protect carbonyl functionality during synthesis¹. Several procedures for regeneration of carbonyl compounds from semicarbazones have been reported³⁻¹². Although some of these procedures are carried out under mild conditions, many of the methods require rare, hazardous and expensive reagents, drastic reaction conditions or sometimes longer reaction period. Thus, there is still a need to develop a new, mild, inexpensive and facile procedure for the regeneration of carbonyl compounds from semicarbazones.

Sodium perborate (SPB) is a very cheap, large scale industrial chemical which is extensively used in the detergent industry as well as bleaching and antiseptic agent. Consequently, the ability of SPB to release oxidative species in an organic medium has made it useful reagent in organic synthesis¹³. McKillop *et al*¹⁴ have used SPB for efficient regeneration of carbonyl compounds from hydrazones.



We now report herein an exceptionally simple and efficient desemicarbazonation reaction using sodium perborate.

The semicarbazones of aromatic and aliphatic aldehydes and ketones react smoothly with SPB under mild conditions to give corresponding aldehydes and ketones in almost quantitative yields. The procedure is simple and convenient. In addition the reagent, SPB used is inexpensive, nonhazardous and easily available.

Experimental

General procedure - A mixture of semicarbazone (aldehyde or ketone) (3 mmoles), gl. acetic acid (20 mL) and sodium perborate (7.5 mmoles) was stirred at 40° C for the specified time period (Table I). The reaction was monitored on TLC. After completion of the reaction, the product was extracted with ether and washed with 10% sodium bicarbonate and water. The ether extract was dried (anhyd. Na₂SO₄) and the solvent removed under reduced pressure to afford the corresponding aldehyde/ketone in excellent yields (95-97%).

Table I—Oxidative cleavage of semicarbazones with sodium perborate

Semicarbazones of	Reaction period (hr)	Semicarbazones of	Reaction period (hr)
Butyraldehyde	3.5	Cyclopentanone	2.2
4-Methylpentan-2-one	1.5	Cyclohexanone	3.0
Ethyl acetoacetate	1.5	Acetophenone	0.7
Furfuraldehyde	1.0	<i>p</i> -Methylacetophenone	0.6
Benzaldehyde	1.0	<i>p</i> -Chloroacetophenone	2.6
<i>p</i> -(<i>N,N</i> -dimethylamino)-benzaldehyde	1.5	Benzophenone	3.0

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[†]For Part 3, see ref. (1)

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